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(54)Oxygen production by adsorption

A process for producing an oxygen enriched product includes: (a) providing a gas separation apparatus having at least one bed containing a mixture of at least two different nitrogen selective adsorbents, wherein the at least one bed is free of lithium cations; (b) feeding a feed gas containing oxygen and nitrogen into the gas separation apparatus to contact the at least one bed; and (c) recovering from the gas separation apparatus the oxygen enriched product. The process is preferably performed above ambient temperature and/or in a simplified four-step cycle. The cycle includes: (a) feeding a feed gas containing oxygen into a gas separation apparatus to contact at least one bed of the apparatus with the feed gas, wherein the feed gas is at a temperature above ambient; (b) countercurrently evacuating the at least one bed following the feeding; (c) countercurrently purging the at least one bed with the oxygen enriched product under vacuum; and (d) simultaneously pressurizing the at least one bed with a countercurrent stream of the oxygen enriched product and a cocurrent stream of the feed gas.

Description

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BACKGROUND OF THE INVENTION

[0001] This invention relates generally to the separation of oxygen from gas mixtures, such as air. More particularly, the invention relates to the use of mixed adsorbents in the adsorber, a higher air feed temperature, and/or a simpler vacuum swing adsorption/pressure swing adsorption (VSA/PSA) process design to separate oxygen from air.

[0002] In numerous chemical processing, refinery, metal production and other industrial applications, purified gas streams are employed for a variety of processing purposes. For example, high purity oxygen is used in chemical processing, steel mills, paper mills, and in lead and gas production operations. Oxygen and nitrogen are produced from air, typically by cryogenic distillation for large size applications. While such cryogenic processing can be very efficient, particularly when conducted in large size plants, it nevertheless requires complex and costly equipment.

[0003] Vacuum swing adsorption/pressure swing adsorption processes have also been used to separate and purify gases, but the production of oxygen by the VSA/PSA approach has generally been confined to relatively small-sized operations with respect to which the use of cryogenic air separation may not be economically feasible. Many commonly available adsorbents, particularly the class of materials known as molecular sieves or zeolites, selectively adsorb nitrogen more strongly than oxygen, and this preferential adsorption is the basis of a variety of VSA/PSA processes that have been developed for the separation of air to produce oxygen and nitrogen product gas.

[0004] In the VSA/PSA process, a feed gas mixture, such as air, containing a more readily adsorbable component and a less readily adsorbable component, e.g., the nitrogen and oxygen components of air, is passed to the feed end of an adsorbent bed capable of selectively adsorbing the more readily adsorbable component at a higher adsorption pressure. Most of the less readily adsorbable component passes through the bed and is recovered from the discharge end of the bed. Thereafter, the bed is depressurized to a lower desorption pressure for desorption of the more readily adsorbable component, and its removal from the feed end of the bed prior to repressurization with feed gas and or less readily adsorbed component, and introduction of fresh feed gas for adsorption as cyclic adsorption-desorptionrepressurization operations are continued in the bed. Such VSA/PSA processing is commonly carried out in multi-bed systems, with each bed employing the same VSA/PSA processing sequence on a cyclic basis interrelated to the carrying out of such processing sequence in the other beds of the adsorption system. In VSA/PSA systems for the recovery of moderate to high purity oxygen (80-95% O2) product as the less readily adsorbable component of air, each adsorbent bed will commonly contain an adsorbent material capable of selectively adsorbing nitrogen as the more readily adsorbable component, with the selectively adsorbed nitrogen being subsequently desorbed and removed from the bed upon reduction of the pressure of the bed from the higher adsorption pressure level to a lower desorption pressure level. VSA/PSA systems for the recovery of nitrogen product have likewise been based on the use of adsorbents that selectively adsorb nitrogen from air as the more readily adsorbable component thereof.

[0005] There are various techniques that exist to separate nitrogen from oxygen. For instance, U.S. Pat. No. 4,329,158 to Sircar discloses a process for the separation of nitrogen from oxygen wherein a pretreatment adsorptive separation of water and carbon dioxide is performed prior to the bulk separation of the major constituents of air. Nitrogen enriched waste gas is utilized from the bulk separation portion of the process to regenerate the pretreatment portion of the process. The bulk separation of nitrogen from oxygen is performed with an elevated temperature adsorption of nitrogen, a desorption of bulk separation beds to a lower pressure, a purge of the beds with product oxygen after desorption countercurrently and two steps of repressurization to elevated pressure first with waste gas which is nitrogen enriched and secondly with product oxygen.

[0006] U.S. Pat. No. 5,882,380 to Sircar describes a single-bed PSA system comprising a blower, an adsorber vessel, and a gas product storage tank that separates a gas mixture using a three-step cycle comprising adsorption, evacuation, and pressurization used to separate nitrogen from a feed air. Pressurization is accomplished by introducing gas from the gas product storage tank into both the feed end and the product end of the adsorber vessel. Preferably a portion of the pressurization gas is introduced into the adsorber vessel by the blower, which also is used for providing feed to the adsorber and for withdrawing gas from the adsorber during the evacuation step.

[0007] Ackley et al. (European Patent Application No. 0 963 777) discloses a PSA apparatus for the separation of a heavy component from a light component in a feed stream. The apparatus includes an adsorbent bed comprising either a mixture of adsorbents or composite adsorbent particles wherein each particle comprises two or more adsorbents. At least one of the adsorbents is comparatively weak, i.e., NaX, and the other is comparatively strong, i.e., LiX. Another embodiment of the invention is a PSA prepurifier having a bed of adsorbent material which comprises a mixture of adsorbents, or composite of adsorbent particles wherein each particle comprises at least two adsorbents, at least one of the adsorbents being comparatively strong, i.e., NaY and at least another of the adsorbents being comparatively weak, i.e., activated aluminum.

[0008] The adsorbent is often the key to the effectiveness of oxygen production processes. Therefore, much attention has been given to the development, improvement and manufacture of adsorbents. For example, specialized zeolite

adsorbents have been synthesized through ion exchange, lower Si/Al structures and improved activation procedures. These additional and/or improved manufacturing steps have resulted in higher costs for these specialized adsorbents (e.g., LiX) compared to more common adsorbents (e.g., 5A and 13X). In many processes, the adsorbent has become a significant fraction of the overall capital investment. Thus, there is considerable incentive to reduce the cost of the adsorbent if doing so results in an overall reduction in the cost of the desired product of the separation.

[0009] Accordingly, there is a need for alternative systems for separating oxygen from gas mixtures, such as air, wherein the systems optimize the productivity of relatively inexpensive adsorbents to render the systems economically competitive with state of the art systems employing more sophisticated but expensive adsorbents.

[0010] All references cited herein are incorporated herein by reference in their entireties.

BRIEF SUMMARY OF THE INVENTION

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[0011] The invention provides a process for producing an oxygen enriched product from a feed gas containing oxygen and nitrogen. The process comprises: (a) providing a gas separation apparatus having at least one bed containing a physical mixture of at least two different nitrogen selective adsorbents, wherein the at least one bed is free of lithium cations; (b) feeding a feed gas containing oxygen and nitrogen into the gas separation apparatus to contact the at least one bed; and (c) recovering from the gas separation apparatus the oxygen enriched product. The process is preferably performed above ambient temperature and/or in a simplified four-step VSA/PSA cycle. The cycle includes: (a) feeding a feed gas containing oxygen and nitrogen into a gas separation apparatus to contact at least one bed of the apparatus with the feed gas, wherein the feed gas is at a temperature above ambient, e.g., from about 40°C to about 100°C; (b) countercurrently evacuating the at least one bed following the feeding; (c) countercurrently purging the at least one bed with a countercurrent stream of the oxygen enriched product and a cocurrent stream of the feed gas.

[0012] Also provided are an apparatus for performing the process of the invention and a process for producing an oxygen enriched product from a feed gas containing oxygen and nitrogen using the four-step cycle and elevated temperature with lithium-containing and/or lithium-free beds.

BRIEF DESCRIPTION OF SEVERAL VIEWS OF THE DRAWINGS

[0013] The Invention will be described in conjunction with the following drawings wherein:

Figs. 1, 2 and 3 are graphs of N₂ capacity versus adsorption pressure.

DETAILED DESCRIPTION OF THE INVENTION

[0014] While various techniques exist to separate nitrogen from oxygen, the inventors are not aware of the use of mixed adsorbents in the adsorber column which completely exclude the use of a lithium exchange cation, and higher feed temperature, or simpler process designs.

[0015] There is a need for a vacuum swing adsorption/pressure swing adsorption (VSA/PSA) system and process that reduces the cost of adsorption processes. This need can be met by improving adsorbent efficiency and/or by reducing the cost of the adsorbent. Improved adsorbent efficiency means greater adsorbent effectiveness for the desired separation.

[0016] While conventional zeolite molecular sleves associated with lithium exhibit a highly desirable capacity and selectivity toward the adsorption of nitrogen from air, it has been found that less costly mixtures of at least two low to moderate nitrogen selective adsorbents, such as zeolites containing monovalent and divalent cations which include calcium, sodium, barium, strontium, magnesium, and cesium, can also be used for the desired selective adsorption of nitrogen from feed air, and the recovery of oxygen as the desired product gas at a reduced or comparable cost. Other low cost nitrogen selective zeolitic adsorbents suitable for use in the present invention are exchanged X, exchanged A, and mordenite zeolites, such as NaX, 5A, Na-Mordenite, CaX and CaLSX.

[0017] In preferred embodiments, the separation of nitrogen from oxygen is achieved through the use of a mixture of at least two different low to moderate nitrogen selective adsorbents mixed together. The term "mixture" as used herein means a physical intermixture of ingredients (e.g., a homogeneous blend of adsorbent particles) as opposed to a heterogeneous assembly of ingredients (e.g., layers of different adsorbents) inside the adsorber. The mixture can be a composite of adsorbent particles or a mixture of independent adsorbent particles. The mixture preferably contains from about 20% to about 80% by weight of a first adsorbent and from about 80% to about 20% by weight of a second absorbent. The mixture more preferably contains about 50% by weight of each of the two adsorbents. It has been found that the use of mixed adsorbents, instead of a single adsorbent, can produce oxygen at a competitive cost.

[0018] The mixture is preferably free of lithium ion exchanged zeolites, such as LIX. Accordingly, the bed is preferably

free of lithium cations.

[0019] Additionally, it has been found that the use of a higher feed air temperature than ambient, e.g., from about 40°C to about 100°C, can significantly alter the air separation process performance in order to produce cost-competitive oxygen product using relatively inexpensive adsorbents. This method can also be used in conjunction with a relatively simple process design (i.e., a four-step cycle), which results in a single adsorber, single air blower-vacuum pump combined system which produces oxygen at a competitive cost.

[0020] The invention will be illustrated in more detail with reference to the following Examples, but it should be understood that the present invention is not deemed to be limited thereto.

10 EXAMPLES

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[0021] Simulations were performed to study the cost of O_2 production by VSA using pure gas adsorption data for N_2 and O_2 on various zeolites. The relative costs were developed for a 60 TPD contained O_2 unit producing 90-92% O_2 at a pressure of 10 psig. The inventors evaluated the process performance of (a) several single zeolites (NaX, CaX, 5A, Na-mordenite, CaLSX and LiX), (b) several mixed zeolites (LiX + NaX, CaX + NaX) where an intimate mixture of zeolites at the particle level (not layered) was used, and (c) operating the VSA- O_2 process using a hot feed air in order to raise the average adsorbent temperature during the process steps. The commonly used eight-step VSA O_2 process (minimum two beds) as well as a simpler four-step VSA- O_2 process using a single bed and a single air blower/vacuum pump combine were evaluated.

[0022] As a result of this evaluation, it was determined that LiX zeolite offers the best performance for O_2 production under commonly used VSA- O_2 process design conditions (highest O_2 productivity and lowest power) due to its high N_2 working capacity and selectivity and low N_2 Henry's Law constant and that the performance advantage of LiX zeolite is compromised by its relatively higher cost. All other zeolites yielded competitive O_2 product costs when used with the commonly used VSA- O_2 process because of their relatively lower costs. It was also determined that a mixed adsorbent system allows alteration of effective N_2 and O_2 adsorption characteristics by design, which in turn provides large flexibility in process performance. These systems are also cost competitive with LiX based systems.

[0023] Table 1 illustrates examples of the adsorptive properties of various ion-exchanged zeolites of different framework structures (commercially developed) used for practical air separation processes. High nitrogen working capacity, high nitrogen selectivity over oxygen, and low heat of adsorption of nitrogen generally lower the specific adsorbent inventory for a given oxygen production capacity and give higher oxygen recovery from the feed air. Lower Henry's Law constants for nitrogen and lower heats of adsorption for N₂ generally reduce the evacuation power for desorption of N₂ in a VSA-O₂ process.

[0024] Table 1 lists adsorbents in chronological order of development of zeolites for air separation. The N_2 working capacity increases and then levels off, the selectivity of adsorption of N_2 over O_2 increases and then levels off, and the heat of adsorption of N_2 increases and then levels off. On the other hand, the Henry's Law constant for N_2 increases and then decreases for LiX. Thus, LiX is currently the preferred material for production of O_2 because it provides the most desired properties. However, LiX is also the most expensive adsorbent listed.

Table 1

Example No.	Adsorbent	Relative Cost	N ₂ Working Capacity	N ₂ /O ₂ Selectivity	N ₂ Isosteric Heat	N ₂ Henry's Law Constant	
		\$/Ib	moles/kg		Kcal/mole	moles/kg/ atm	
1	NaX	1.00	0.110	2.9	4.3	0.297	
2	5A .	1.07	0.170	3.8	5.7	0.522	
3 .	Na-Mordenite	2.67	0.216	4.0	6.2	0.982	
4	CaX	1.27.	0.252	10.9	7.1	3.606	
5	CaLSX	2.33	0.387	12.7	6.9	4.033	
6	LiX	3.70	0.342	10.2	6.7	1.982	

*Isothermal N_2 working capacity at 30°C: Adsorption of air at 1 atm Evacuation to 0.3 atm

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[0025] The performance of a commonly used VSA- O_2 process for production of 90-92% oxygen from ambient air using the zeolite adsorbents of Table 1 was simulated using experimentally measured N_2 and O_2 adsorption characteristics on these materials. The commonly used VSA- O_2 process consisted of eight cyclic steps: (a) feed air flow with O_2 enriched product withdrawal, (b) feed air flow to provide O_2 enriched purge gas, (c) cocurrent depressurization to provide purge gas, (d) cocurrent depressurization to provide pressurization gas, (e) countercurrent evacuation, (f) countercurrent purge with O_2 enriched gas under vacuum, (g) countercurrent pressurization with O_2 enriched gas and (h) cocurrent feed pressurization.

[0026] Table 2 shows the simulated separation performances for different zeolites. It may be seen that the O_2 productivity (mlb moles of O_2 produced/lb of zeolite/cycle) and the specific power for the product (KW/TPD contained O_2) substantially vary from zeolite to zeolite, but the relative costs of oxygen product (\$/ton) is very insensitive to the choice of the adsorbents.

[0027] The results in Table 2 illustrate that the LiX zeolite has the highest oxygen production capacity and the lowest power requirement, but its higher price compromises these apparent advantages to give similar oxygen cost as other zeolites. Also, as evidenced by Table 2, other materials, such as NaX or CaX, compete well with LiX because they are less expensive than LiX.

Table 2

Example No.	Adsorbent O ₂ Productivity O ₂ Recovery Relative Special Power*		Relative Specific Power*	Relative O ₂ Cost	
		(mib moles/lb/cycle)	(%)	(KW/TPDc)	
1	LiX	0.0597	70.0	1.00	1.00
2	NaX	0.0304	52.5	1.18	1.04
3	CaX	0.0371	59.4	1.18	1.03
4	CaLSX	0.0495	64.8	1.09	1.01

^{*} Adsorption Pressure = 1.35 atm,

[0028] The inventors noted that even though N_2 adsorption isotherms are strikingly different on these zeolites (as shown by Fig. 1), such disparity in isotherm shapes does not create any meaningful difference in the O_2 production cost using today's zeolite cost structure.

[0029] The above unexpected results led the inventors to invent (a) several different ways of altering the N_2 and O_2 adsorption isotherm shapes and thereby obtain O_2 production costs which are competitive with the current cost for the commonly used VSA- O_2 process using the LiX adsorbent, and (b) several different ways of operating simpler and less capital intensive VSA- O_2 processes than the present complex VSA process in order to match the current cost of O_2 production.

[0030] First, the Inventors discovered that the effective shapes of N₂ and O₂ adsorption isotherms can be altered by design using a packed bed of intimately mixed (not layered) particles of two or more different zeolites. This will introduce a synthetic adsorbent heterogeneity and change the effective adsorption isotherms, N₂ selectivities over O₂ and heats of adsorption for the air separation process. For example, the pure gas adsorption isotherms of a composite adsorbent bed will be given by the weighted averages [(weight % of a type) X (adsorption capacity of that type)] of the isotherms of each type of adsorbent present in the mixture.

[0031] Fig. 2 shows the composite N_2 isotherms for 50% LiX + 50% NaX and 50% CaX + 50% NaX mixtures. As illustrated in Fig. 2, drastic changes in isotherm shapes can be created (compared to single adsorbents) by mixing different adsorbents. It was also observed that the corresponding unit cost of the mixed adsorbent is significantly less than that of single adsorbents.

[0032] Table 3 illustrates the simulated performance of a commonly used VSA-O₂ process using the above described mixed adsorbents. The operating conditions of the processes are the same as those used for generating the data of Table 2. It may be seen that even though the mixed gas adsorbents exhibit lower O₂ productivity and higher specific power, the O₂ production cost is comparable with that of LiX due to the less expensive cost of NaX and CaX zeolites.

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Final Desorption Pressure = 0.323 atm

Feed Temperature = 97°F, Product compressed to 10 pslg

Table 3

Example No.	Adsorbent	O ₂ Productivity	O ₂ Recovery	Relative Specific Power*	Relative O ₂ Cost	
		(mibmoles/lb/ cycle)	(%)	(KW/TPDc)		
1	LiX	0.0597	70.0	1.00	1.00	
2	50%LiX+50%NaX	0.0491	65.0	1.02	0.99	
3	50% CaX+ 50% NaX	0.0353	57.3	1.16	1.02	

^{*} Same operating conditions as those for Example 2.

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[0033] Secondly, the inventors have discovered that the shapes of the N₂ and O₂ adsorption isotherms can also be altered by changing the effective adsorbent temperature within the adsorbers during the air separation process steps. This can be achieved by increasing the feed air temperature above ambient, e.g. from about 40°C to about 100°C. [0034] Fig. 3 shows the N₂ adsorption isotherm of CaLSX zeolite at 140°F (60°C) and compares it with the N₂ isotherm of LiX at 30°C. As illustrated by Fig. 3, the Henry's Law constant of CaLSX becomes similar to that of LiX (30°C) at 60°C. Table 4 demonstrates the performance of a commonly used VSA-O₂ process at 140°F (60°C) feed temperature using CaLSX. CaLSX is a highly nitrogen selective binderless exchange X zeolite adsorbent. At a temperature above ambient, e.g., from about 40°C to about 100°C, the inventors discovered that the process performance and O₂ costs of CaLSX are comparable with those for LiX.

Table 4

Example No.	Adsorbent	Feed Gas Temperature	O ₂ Productivity	O ₂ Recovery	Relative Specific Power*	Relative 0 ₂ Cost
		(°F)	(mlbmoles/lb/ cycle)	(%)	(KW/TPDc)]
1 .	LiX	97.0	0.0597	70.0	1,00	1.00
2	CaLSX	140.0	0.0557	68.9	1.03	0.98
3	50%CaX+ 50% NaX	140.0	0.0339	58.0	1.16	1.03

^{*} Same operating conditions as used for the cases of Table 2 except the feed gas temperature.

[0035] The data in Table 4 indicate that the effective N₂ and O₂ adsorption characteristics can also be altered by using higher temperature (above ambient) air feed to the VSA-O₂ system. This approach can be used to obtain process performance which is very similar to that for LiX by using less expensive adsorbents. This option may be particularly attractive because the currently required feed gas cooling step after the air blower used in a single bed VSA system using a highly selective nitrogen adsorbent can be eliminated.

[0036] Additionally, concepts described above in Tables 3 and 4 (i.e., intimately mixed adsorbents and high feed temperature) can be used in conjunction with a simpler VSA cycle which eliminates the need for the use of multiple beds. Thus, a single bed, single blower-vacuum pump combine can make the process simpler yet cost effective. The impact of changing materials and process design on the cost of O₂ product are not significant partly because (a) higher O₂ productivity generally accompanied by higher power which compensate each other and (b) adsorbents which give better performance are also more expensive which neutralizes the performance advantage. Thus, less expensive commercial adsorbents can be used for competitive O₂ cost production when used with a discreet process design.

[0037] A concept for economic production of 80-95% oxygen from air was developed by using a VSA process cycle where a combination (physical mixture or composite) of two or more nitrogen selective adsorbents (having low to moderate nitrogen adsorption selectivity and capacity) used in conjunction with a variety of process cycle designs and elevated air feed gas temperatures. The net oxygen product costs from such a system is lower than or equivalent to that obtained by using an expensive high performance adsorbent, such as LIX, with high nitrogen adsorption selectivity and capacity.

[0038] The above described use of mixed adsorbents with or without higher feed air temperature in changing the

effective shape of the N2 and O2 isotherms can also be used in conjunction with simple (four-step cycle) VSA-O2 processes for lowering the cost of O2 production. The eight-step commonly used VSA-O2 process requires at least two parallel adsorbent beds in tandem in order to provide the Internal purge and pressurization gases. The simpler four-step VSA-O2 process for producing an O2 enriched product comprises: (a) feeding a feed gas (preferably air) into a PSA or VSA bed; (b) countercurrent evacuation of said bed, (c) countercurrent purge of said bed with O2 product under vacuum, and (d) simultaneous pressurization of sald bed with O2 product (countercurrent) and feed air (cocurrent). This simpler four-step cycle process can be operated using a single adsorber and a single combined-air blower and vacuum pump at a lower capital cost, because there are no steps where gas communication between two adsorbers are needed.

[0039] Table 5 shows the comparative performance of the above described simpler four-step cycle VSA-O2 using CaX zeolite at a feed air temperature of 140°F (60°C). The O₂ production costs is very competitive with that for the commonly used VSA-O₂ process using LiX zeolite.

			Table	9 5			
Ex. No.	Process	Adsorbent	Feed Gas Temperature	O ₂ Productivity	O ₂ Recovery	Relative Specific Power*	Relative O ₂ Cost
	·		(°F)	(mibmoles /ib/cycle)	(%)	(KW/TP Cd)	
1	Commonly Used VSA* (8step cycle)	LIX	97.0	0.0597	70.0 ⁻	1.00	1.00
2	Simple-VSA** (4-step cycle)	CaX	140.0	0.0337	41.3	1.43	1.026
3	Simple-VSA** (4-step cycle)	50%CaX+ 50%NaX	97.0	0.0317	40.9	1.39	1.04
4	Simple-VSA** (4-step cycle)	50%CaX+ 50%NaX	140.0	0.0291	40.2	1.36	1.06

[0040] The above demonstrates that N_2 and O_2 adsorption isotherms for air separation can be manipulated in various ways by using mixed adsorbents and/or using higher feed air temperature in order to produce a cost effective Op product which is competitive with the commonly used VSA O2 process using LiX zeolite as the adsorbent. This is a surprising result. The flexibility created by the choice of pure or mixed adsorbents and operating conditions can also be utilized with a simpler four-step cycle VSA-O2 process which can be operated using a single adsorber and a single air blower-vacuum pump combination which also results in a competitive oxygen cost.

[0041] The above described results also show that many commercially available and less expensive adsorbents, such as NaX, CaX, and 5A, or their combinations in conjunction with many different VSA-O2 processes of simpler designs and operating protocols can be used to produce an O2 product which competes well with the commonly used VSA-O2 process costs.

[0042] The present invention has been set forth with regard to several preferred embodiments, however the full scope of the present invention should be ascertained from the claims which follow.

Claims

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- 1. A process for producing an oxygen enriched product from a feed gas containing oxygen and nitrogen, said process
- providing a gas separation apparatus comprising at least one bed containing a physical mixture of at least two different nitrogen selective adsorbents,

wherein said at least one bed is free of lithium cations;

feeding said feed gas into said gas separation apparatus to contact said at least one bed; and

^{*} Same operating conditions as those for Table 2.

^{**} Adsorption pressure = 1.35 atm

Final desorption pressure = 0.40 atm

recovering from said gas separation apparatus said oxygen enriched product.

- 2. The process according to claim 1, wherein said feed gas is air.
- The process according to claim 1, wherein said mixture is a composite of adsorbent particles or mixture of independent adsorbent particles.
 - 4. Theprocess according to claim 1, wherein said mixture is a homogeneous mixture of adsorbent particles.
- 5. The process according to claim 1, wherein one of said two different nitrogen selective adsorbents is a zeolite.
 - 6. The process according to claim 5, wherein said zeolite contains a monovalent cation or a divalent cation.
- 7. The process according to claim 1, wherein sald two different nitrogen selective adsorbents are two different members selected from the group consisting of sodium, calcium, barium, strontium, magnesium, cesium, exchanged X zeolite, exchanged A zeolite, and mordenite zeolite.
 - 8. The process according to claim 1, wherein said mixture contains from about 20% to about 80% by weight of a first of said two different nitrogen selective adsorbents, and from about 80% to about 20% by weight of a second of said two different nitrogen selective adsorbents.
 - 9. The process according to claim 7, wherein said mixture contains about 50% by weight of each of said two different nitrogen selective adsorbents.
- 25 10. The process according to claim 1, further comprising providing said feed gas at a temperature above ambient.
 - 11. The process according to claim 10, wherein said temperature is from about 40°C to about 100°C.
 - 12. The process of claim 1, further comprising:

countercurrently evacuating said at least one bed following said feeding; countercurrently purging said at least one bed with said oxygen enriched product under vacuum; and simultaneously pressurizing said at least one bed with a countercurrent stream of said oxygen enriched product and a cocurrent stream of said feed gas.

- 13. The process according to claim 12, further comprising providing said feed gas at a temperature above ambient.
- 14. The process according to claim 13, wherein said temperature is from about 40°C to about 100°C.
- 40 15. A process for producing an oxygen enriched product, said process comprising:

providing a gas separation apparatus comprising at least one bed; feeding a feed gas containing oxygen and nitrogen into said gas separation apparatus to contact said at least one bed, wherein said feed gas is at a temperature above ambient; countercurrently evacuating said at least one bed following said feeding; countercurrently purging said at least one bed with said oxygen enriched product under vacuum; simultaneously pressurizing said at least one bed with a countercurrent stream of said oxygen enriched product and a cocurrent stream of said feed gas; and

- recovering from said gas separation apparatus said oxygen enriched product.
- 16. The process according to claim 15, wherein said temperature is from about 40°C to about 100°C.
- 17. The process according to claim 13, wherein said at least one bed contains a mixture of at least two different nitrogen selective adsorbents, and is free of lithium cations.
- 18. A gas separation apparatus adapted to perform the process of claim 1.
- 19. The gas separation apparatus of claim 18, comprising a single adsorber and a single combined air blower-vacuum pump.

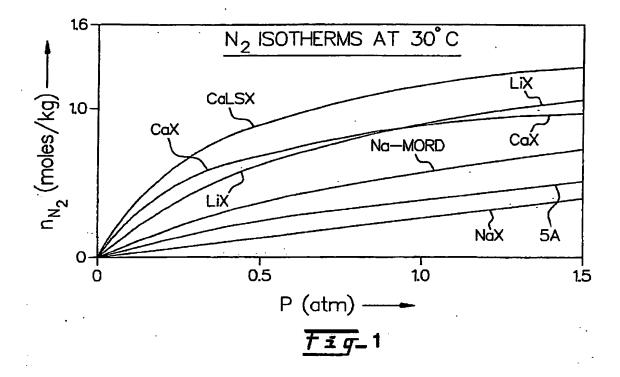
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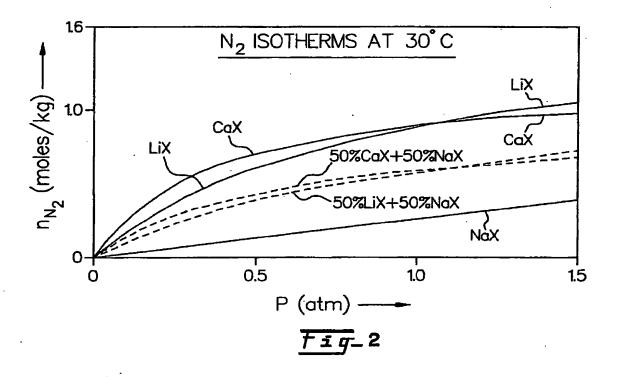
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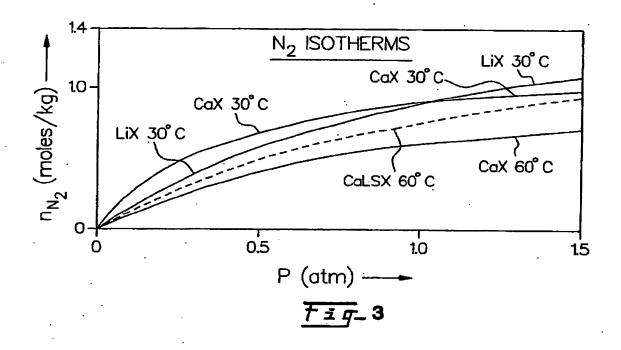
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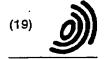
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(54) Oxygen production by adsorption

(57) A process for producing an oxygen enriched product includes: (a) providing a gas separation apparatus having at least one bed containing a mixture of at least two different nitrogen selective adsorbents, wherein the at least one bed is free of lithium cations; (b) feeding a feed gas containing oxygen and nitrogen into the gas separation apparatus to contact the at least one bed; and (c) recovering from the gas separation apparatus the oxygen enriched product. The process is preferably performed above ambient temperature and/or in a simplified four-step cycle. The cycle includes: (a) feed-

ing a feed gas containing oxygen into a gas separation apparatus to contact at least one bed of the apparatus with the feed gas, wherein the feed gas is at a temperature above ambient; (b) countercurrently evacuating the at least one bed following the feeding; (c) countercurrently purging the at least one bed with the oxygen enriched product under vacuum; and (d) simultaneously pressurizing the at least one bed with a countercurrent stream of the oxygen enriched product and a cocurrent stream of the feed gas.



EUROPEAN SEARCH REPORT

Application Number EP 01 12 9302

	DOCUMENTS CONSIDE	RED TO BE RELEVAN	r		
Calegory	Citation of document with inc of relevant passa	lication, where appropriate. ges	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.CI.7)	
X	EP 1 004 342 A (AIR CHEMICALS) 31 May 20 * the whole document	00 (2000-05-31)	1-19	B01D53/04 B01D53/047 C01B13/02	
x	EP 1 004 341 A (AIR CHEMICALS) 31 May 20 * the whole document	00 (2000-05-31)	1-19		
x	EP 1 027 914 A (AIR CHEMICALS) 16 August * the whole document	2000 (2000-08-16)	1-19		
				• ,	
}				TECHNICAL FIELDS SEARCHED (Int.CI.7)	
				B01D C01B	
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	The present search report has bee				
	Place of search	Date of completion of the search		Examiner	
7	HE HAGUE	17 March 2003	Boga	erts, M	
X : particu Y : particu docum A : techno O : non-w	EGORY OF CITED DOCUMENTS liarly relevant if taken alone larly rothwant if combined with another ent) of the same category logical background ritten disclosure ediate document	E : earlier patent d after the filing d D : document cited L : document cited	T: Iheory or principle underlying the invite: E: earlier patent document, but publishe after the filing date D: document cated in the application L: document cated for other reasons &: member of the same patent lamily, or		

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ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO.

EP 01 12 9302

This annex lists the patent lamily members relating to the patent documents cited in the above—mentioned European search report. The members are as contained in the European Patent Office EDP file on The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

17-03-2003

Patent document cited in search report		Publication date		Patent family member(s)		Publication date
EP 1004342	A	31-05-2000	US BR EP EP JP JP KR TW	9905720 1004342 1018359 3310249 2000157828	A A A2 A2 B2 A A B	14-11-2000 29-08-2000 31-05-2000 12-07-2000 05-08-2002 13-06-2000 25-08-2000 28-05-2001
EP 1004341	A	31-05-2000	US US EP EP	6102985 6096115 1004341 1018359	A A2	15-08-2000 01-08-2000 31-05-2000 12-07-2000
EP 1027914	Α	16-08-2000	US BR EP TW	6183538 0000252 1027914 467759	A A2	06-02-2001 26-09-2000 16-08-2000 11-12-2001

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